

Application No. SS60-58003, filed March 22, 1985; Inventors: Susumu NAGASHIMA, and Kanji SHIMIZU; Assignee: Mitsubishi Chem. Ind. Ltd.

In more detail, specifically, it concerns a dye composition of a reactive disperse dye that uses a non-ionic surfactant and an organic acid salt.

Prior Art

In Prior Art, in the dyeing of a mixed cloth made of polyester fibers and cellulose fibers, dispersive dyes were used for polyester fibers, while reactive dyes, indanthrene dyes, and sulfide dyes etc. were used for cellulose fibers. With this problem in mind, matching the two dye colors for dyeing was extremely hard; moreover, another problem was that of inadequate dye reproducibility.

Recently, dyeing a mixed cloth of polyester fibers and cellulose fibers with a reactive disperse dye has become possible (the 13th Fiber Processing Symposium, November 9, 1984, sponsored by Fiber Science Association). This has solved the problems of color matching and dye reproducibility of Prior Art, while at the same time it became possible to dye cloth in a variety of colors without restricting the application of dye.

However, the dyeing property in continuous dyeing using reactive disperse dye is poor, thus causing the problems of dye fastness and uniform dyeing.

Problems to be Solved by Invention.

The purpose of this invention is to offer a dye composition of a reactive dispersive dye that could continuously [be used to] dye mixed cloth of polyester fiber and cellulose fiber continuously in a single bath and a single stage process whereby the dyeing propertybhjk[n would be increased, and the dye's fastness uniformity and reproducibility would be improved, due to the use of a specific non-ionic surfactant and an organic acid salt.

The means of solving these problems are that this invention is that this invention is a dye composition that includes 0.5 ~ 30 weight parts reactive disperse dye that is insoluble or difficultly soluble in water and 2.5 ~ 15 weight % nonionic surfactant expressed by formula (I)



(in the formula, n designates 6 ~ 30), and moreover, its pH is adjusted to 6.5 ~ 7.5 using an organic acid salt expressed by the general formula (II)



(in the formula, R stands for hydrogen atom, an aliphatic group or an aromatic group with 1 ~ 6 carbon atoms, X stands for a hydroxyl group, or an amino group, and M stands for NH_4 , K, Na; moreover, a is 0 ~ 1, b is 0 ~ 2, c is 0 ~ 2).

The reactive dispersive dye that is insoluble or difficultly soluble in water is, for example, expressed by the general formula (III)



(in the formula, D stands for a pigment group that is insoluble in water, X stands for an oxygen atom or a -NH- group, Y stands for OR³ or NR⁴R⁵, R³, R⁴, and R⁵ stand for a hydrogen atom or for an alkyl group, an aryl group or an alkyl group that may have a

substituting group, or it stands for an nitrogen-including heterocyclic ring of 5 or 6 members formed by bonds between R⁴ and R⁵.)

Examples of D can include compounds of anthraquinone origin, azo origin, quinophthalone origin, methane origin, and heterocondensed cyclic origin.

The amount of the reactive dispersive dye used in the dye composition of this invention can be exemplified in the range between 0.5 ~ weight%, preferably it is in the range of 2.5 ~ 20 weight %.

The nonionic surfactant shown in the general formula (I) can be easily prepared by conventional methods, e.g., by the addition reaction of methanol and ethylene oxide.

Moreover, the number n of added moles of ethylene oxide in the general formula (I) of this invention can be within the range of 7 ~ 10 . Moreover, its amount used can be within the range of 2.5 ~ 15 weight %, preferably, 5.0 ~ 10 weight %.

The organic acid salt expressed by the general formula (II) above can be one of the NH₄, Na and K salts of the organic acids exemplified below:

Formic acid	HCOOH
Acetic acid	CH ₃ COOH
Benzoic acid	C ₆ H ₅ COOH
Phthalic acid	C ₆ H ₄ (COOH) ₂
Lactic acid	CH ₃ CH(OH)COOH
Oxalic acid	(COOH) ₂
Succinic acid	(CH ₂ COOH) ₂
Tartaric acid	$\begin{array}{c} \text{HO} - \text{CH} - \text{COOH} \\ \\ \text{HO} - \text{CH} - \text{COOH} \end{array}$
Citric acid	$\begin{array}{c} \text{CH}_2 - \text{COOH} \\ \\ \text{HO} - \text{C} - \text{COOH} \\ \\ \text{CH}_2 - \text{COOH} \end{array}$

Ammonium carbamate H₂NCOOH

Furthermore, the pH adjustment in the general formula (II) of this invention is exemplified within the range of 6.5 ~ 7.5. The preferred range is 6.5 ~ 7.5.

The dye composition of this invention is obtained by dispersing/dissolving the said reactive disperse nonionic surfactant of the general formula (I) and the organic acid salt of the general formula (II) in water.

Furthermore, the dye composition of this invention is obtained by dispersing/dissolving the said reactive disperse nonionic surfactant of the general formula (I) and the organic acid salt of the general formula (II) in water, and the amount of said water used is more than 50% by weight, preferably 70 ~ 95% by weight.

In addition, to the dye composition of this invention can be added, without any harm, 15% by weight or less of impregnating – wetting agents, hydrotropic agents, migration preventing agents and other adjuvants. Impregnating-wetting agents may include Daiasaba-PM-SR (the trade name of a product manufactured by Mitsubishi Kasei Kogyo K.K.), Succinol CS (the trade name of a product manufactured by Nihon Nenka Kogyo K.K.), Tecsupport D-24 (the trade name of a product manufactured by Nika Kagaku Kogyo), etc.

Hydrotropic agents include urea, dimethyl urea, sodium toluene sulfonate, etc. Migration preventing agents include Daiasaba-MG-N (the trade name of a product manufactured by Mitsubishi Kasei Kogyo K.K.), Damanori SA-25 (Daiasaba-PM-SR (the trade name of a product manufactured by Arakawa Kagaku Kogyo K.K.), Mignon NS (Daiasaba-PM-SR (the trade name of a product manufactured by Nihon Nenka Kogyo K.K.), Dakkuarugin NSPM (Daiasaba-PM-SR (the trade name of a product manufactured by Toshifumi Company Ltd.), etc.

Practical Examples

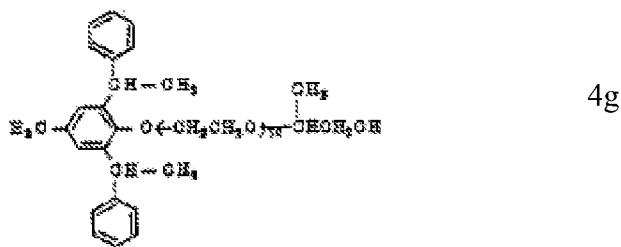
Next, we will provide specific explanations based on Practical Examples of this invention; however, this invention is not limited to these Practical Examples.

The preparation of the reactive dispersive dye in various Practical Examples and Comparison Examples and the various evaluation methods are as follows.

(1) Preparation of the reactive dispersive dye:

A mixture of the following composition

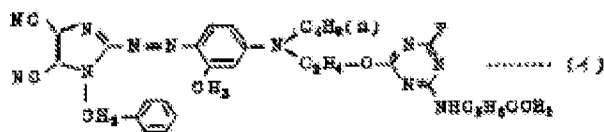
Reactive dispersive dye 15 g
the following structural formulas (a), (b) or (c)



Lignin sulfonate	3 g
(Manufactured by Westvaco, trademark Reax 85A	
Ethylene glycol	5 g
Water	73 g
Total	100 g

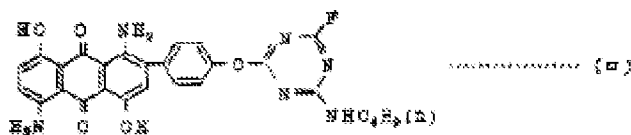
is pulverized at room temperature in a sand grinder and a dye dispersion liquid is prepared.

(a)



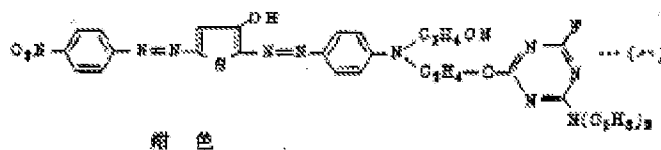
Red color

(b)



Blue-green color

(c)



Navy blue color

(2) Continuous dyeing method

The pre-determined dyeing composition was prepared and made into a pad bath. Using it, a cloth with a 65 : 35 mixed waving ratio of polyester fibers to cellulose fibers was dyed continuously. The process and conditions were as follows.

Padding /dip - / nip Contraction percentage 65%

↓

Drying 100°C x 130° sec

↓

Dry heat stabilizing 210°C x 60 sec

↓

Rinsing

↓

Washing clean 2 g/l sodium carbonate, 2 g/l Daisaba SC/CT

↓

Drying 100°C x 120 sec.

(3) Evaluation of the dyeing property

The surface reflection rate of the dyed cloth resulting from (1) as described above was measured with a color difference meter (manufactured by Nihon Denshiki Kogyo K.. K.) and relative values were calculated assuming the surface reflection factor of the dyed cloth resulting from a comparison example as 100 (benchmark).

(4) Evaluation of fastness

(a) Light Exposure Resistance

Based on JIS Standard L0842-1971, 1/1 N concentration dyed cloth was used and its discoloration was evaluated after exposure to carbon arc for periods of 20 hours and 40 hours

(b) Friction

Based on JIS Standard L0849-1971, 1/1 N concentration dyed cloth was used, and its friction contamination on a lignin cloth with 100% moisture content was evaluated using a [text missing] vibration friction tester.

(5) Evaluation of Uniform Dyeing Property

Using 1/1N concentration dyed cloth, dyeing uniformity (stripes, spots, etc.) was evaluated by relative comparison to a dyed cloth obtained in a comparison example.



Excellent Uniformly dyed Poor

Practical Example 1.

A dye composition consisting of the following formulation

Dye dispersion liquid [(a)]	5 g
$\text{CH}_3\text{O}-(\text{CH}_2\text{CH}_2\text{O})_7\text{H}$	5 g
Impregnation agent	0.1 g
[Daiasaba PN – SR (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	
Migration prevention agent	0.1 g
[Daiasaba MG - N (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	
Urea	1 g
Water solution of $\text{C}_6\text{H}_5\text{COONa}$	adjusted to pH 7
<u>Water</u>	<u>the rest</u>
Total	100 g

and continuous dyeing described above in (2) was carried out. As a result, it was possible to obtain a uniformly red dyed cloth extremely rich in the dyeing property (concentrated color property).

The comparison of these results in terms of the dyeing property, fastness, and uniformity were compared to Prior Art, which is shown in Table 1.

Practical Example 2

A dye composition consisting of the following formulation

Dye dispersion liquid [(b)]	5 g
CH ₃ O-(CH ₂ CH ₂ O) ₈ H	8 g
Impregnation agent	0.1 g
[Daiasaba PN – SR (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	
Migration prevention agent	0.1 g
[Daiasaba MG - N (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	
Urea	1 g
Water solution of CH ₃ COONH ₄	adjusted to pH 7
Water	<u>the rest</u>
Total	100 g

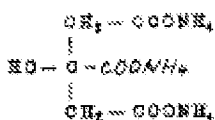
and continuous dyeing described above in (2) was carried out. As a result, it was possible to obtain blue-green dyed cloth with a very good dyeing property (concentrated color property).

The results are shown in Table 1.

Practical Example 3

A dye composition consisting of the following formulation was used

Dye dispersion liquid [(c)]	20 g
CH ₃ O-(CH ₂ CH ₂ O) ₁₀ H	10 g
Impregnation agent	0.1 g
[Daiasaba PN – SR (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	
Migration prevention agent	0.1 g
[Daiasaba MG - N (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	
Urea	1 g
Water solution of	pH adjusted to 7



<u>Water</u>	<u>the rest</u>
--------------	-----------------

Total	100 g
-------	-------

and continuous dyeing described above in (2) was carried out. As a result, it was possible to obtain a navy blue cloth with excellent dyeing property and good uniformity.

The results and the comparison examples are shown in Table 1.

Comparison Example 1

A dye composition consisting of the following formulation

Dye dispersion liquid [(a)]	5 g	
CH ₃ O-(CH ₂ CH ₂ O) ₇ H	5 g	
Impregnation agent	0.1 g	
[Daiasaba PN – SR (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]		
Migration prevention agent	0.1 g	
[Daiasaba MG - N (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]		
Urea	1 g	
Water solution of sodium polyphosphate	}	pH adjusted to 8.3
Water solution of sodium polyphosphate (III)		

<u>Water</u>	<u>the rest</u>
--------------	-----------------

Total	100 g
-------	-------

and continuous dyeing described above in (2) was carried out. At this time a red dyed cloth was obtained with a dyeing property that was worse than in Practical Example 1

The results are shown in Table 1.

Comparison Example 2

A dye composition consisting of the following formulation was used

Dye dispersion liquid [(b)]	5 g
CH ₃ O-(CH ₂ CH ₂ O) ₄ H	7 g
Impregnation agent	0.1 g
[Daiasaba PN – SR (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	

Migration prevention agent	0.1 g
[Daiasaba MG - N (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]	

Urea	1 g
------	-----

Water solution of sodium polyphosphate	} pH adjusted to 8.3
Water solution of sodium polyphosphate (III)	

<u>Water</u>	<u>the rest</u>
--------------	-----------------

Total	100 g
-------	-------

and continuous dyeing described above in (2) was carried out. At this time a blue-green dyed cloth was obtained with poor dyeing property.

The results are shown in Table 1.

Comparison Example 3

A dye composition consisting of the following formulation was used

Dye dispersion liquid [(c)]	20 g
-----------------------------	------

CH ₃ O-(CH ₂ CH ₂ O) ₁₂ H	7 g
---	-----

Impregnation agent	0.1 g
--------------------	-------

[Daiasaba PN – SR (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]

Migration prevention agent	0.1 g
----------------------------	-------

[Daiasaba MG - N (the trademark of a product manufactured by Mitsubishi Kasei Kogyo K.K.)]

Urea	1 g
------	-----

Water solution of sodium polyphosphate	} pH adjusted to 8.3
Water solution of sodium polyphosphate (III)	

<u>Water</u>	<u>the rest</u>
--------------	-----------------

Total	100 g
-------	-------

and continuous dyeing described above in (2) was carried out. At this time a navy blue dyed cloth was obtained with a dyeing property that was worse than in Practical Example 3.

The results are shown in Table 1.

Table 1

			Practical Examples			Comparison Examples		
			1	2	3	1	2	3
Evaluation results	Dyeing Property		210	200	250	std. 100	std. 100	std. 100
	Fastness	Resistance to light (degree)	4	4	4	3	3	3
		Resistance to friction (degree)	2 ~ 3	2 ~ 3	2	1 ~ 2	2	1 ~ 2
	Uniformity		○	○	○	Δ	Δ	Δ

United States Patent and Trademark Office
Translations Branch
Irina Knizhnik
February 26, 2008